

# Flowability of powders for selective laser sintering (SLS) investigated by round robin test

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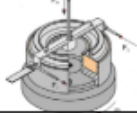


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Many techniques are available to characterize the flow properties of powders. Krantz 2009 provide a comprehensive description of different techniques, considering static and dynamic powder state conditions. Table 1 gives an overview of the most common techniques in use, particularly in the food and pharmaceutical industry.

Table 1. Most common powder characterization methods

Method		Condition	Report
Bed Expansion Ratio		Dynamic under vertical fluid drag load	Fluidized height v/s upstream fluid flow
Angle of repose DIN ISO 4324		Static under free external load	Pile angle
Ring Shear Cell ASTM D 6773		Quasi-Static under pressure	Shear force v/s normal pressure/compression rate
Revolution Powder Analyzer		Dynamic under rotation	Avalanche angle /Surface fractal; Fluidisation
Bulk/Tap Density ASTM D 7481		Static under the effect of powder weight	Loose and packed height v/s n° of taps (Hausner Ratio)

It is well known that results provided by each method are strongly dependent upon the powder stress condition. Techniques that reflect an aired state as the bed expansion ratio are appropriate to predict the fluidization performance, while methods with externally charged and compacted powder give a suggestion about static stability and compressibility ratio. Some of these methods are easy to access and can be simply accomplished (Angle of repose/ Bulk & Tap density). Others need higher sophisticated apparatus and corresponding investments (Ring Shear Cell, RPA). However, presently no specific method has been established for SLS powders. It is not completely clear which one of these methods provides reliable analytical data for SLS powders currently.

In order to reveal the viability of an analytical method for a certain measuring problem regarding repeatability and reproducibility it is a common procedure to perform 'Round Robin Tests' (Interlaboratory comparison).

### 1.3 Round Robin Test / Interlaboratory Comparison

A key problem of almost any analytical method is their validation (Ellison, 2000). One possibility to realize a validation is to execute or participate in Round Robin Tests or Interlaboratory Comparisons (Mandel, 1991). Data, generated from different laboratories on the same sample, afford a lot of valuable information regarding the standard deviation of repeatability and reproducibility of the certain analytical method. The most important factors produce deviations between individual measured results are:

a) the operator, b) the equipment and the analytical instruments, c) the calibration of the equipment and the instruments and d) the environmental effects during the test procedure, i.e. influence of temperature, humidity, light, pollution, etcetera.

The factors mentioned above are mathematically described by calculating the main characteristic interlaboratory results:

- the robust **standard deviation of repeatability**  $s_r$  (factors a), b), c) and d) are identical;  
→ **repeatability conditions**;
- the robust **standard deviation of reproducibility**  $s_R$  (factors a), b), c) and d) are varied;  
→ **reproducibility conditions**.

The source and the significance of these terms used in Round Robin Tests are defined in Figure 2 and are further explained by Affolter, 2001.

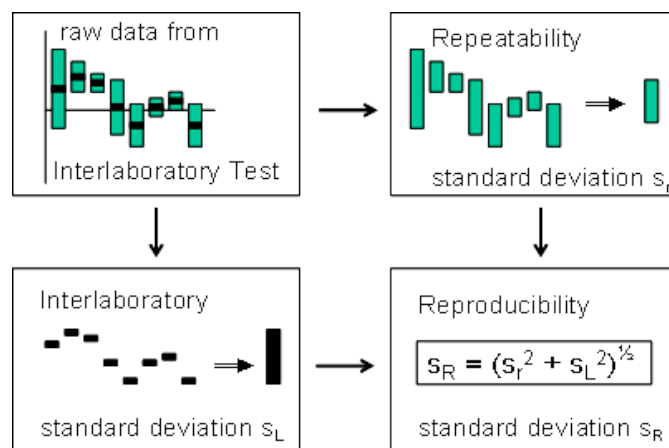


Figure 2. Correlation between statistical terms ( $s_r$ ,  $s_L$  and  $s_R$ ).

The calculations of the presented Round Robin Test are base upon robust statistics as defined in DIN 38402-45. The standard deviations ( $s_r$ ,  $s_R$ ) are calculated iterative. The closer the measured values are at the calculated mean value the higher their weighting. Regarding this statistical model also the mean value (called: 'Hampel estimator') is achieved by iterative calculation. Robust statistics with iterative calculated indicators are of high steadiness as no outlier must be removed during computations and all measured values of all Round Robin Test participants will be incorporated into the results.

## 2 HAUSNER RATIO - ROUND ROBIN TEST

### 2.1 Determination of Hausner Ratio (HR)

The ratio of tap and bulk density provide a statement on the flowability of powders and is called 'Hausner Ratio (HR)'. The following powder classification regarding HR is described from Barbosa-Canovas, 2005.

- $HR < 1.25$  high powder flowability
- $1.25 < HR < 1.4$  decreased powder flowability
- $HR > 1.4$  cohesive powder

SLS powders should be typically under a limit of 1.25 (high flowability) to be regarded as a useful powder with sufficient powder density.

Tap densities are typically obtained with a simple laboratory vibration tamper where an excenter is used for periodical compaction moves (see Table 1). In the present Round Robin Test this concept is even more simplified, as not every participant possesses a respective apparatus defined in ASTM D7481. Thus, for the determination of the Hausner factor in this study, only two laboratory elements are required: a plastic measuring cylinder with a volume of 100 ml and a powder funnel (orifice must match the graduated cylinder) (see Figure 3).



Figure 3. Necessary lab equipment for determination of HR

In total 9 qualified SLS laboratories with sufficient experience in handling SLS powders take part in the Interlaboratory comparison. In order to achieve reliable and compareable information the participant have to adhere to the following instructions strictly:

- Determination of the weight of a 100 ml plastic measuring cylinder ( $m_0$ ). Accuracy of the balance should be at least 0.1 g.
- Fill in the graduated cylinder from a height of approximately 5 cm above the funnel with the experimental powder by pouring it loosely until the 100 ml mark is reached without any mechanical compaction.
- Determination of the total weight (cylinder + powder) ( $m_1$ ).
- Compaction of the powder in the cylinder by hand tapping on a hard surface (table) with low power; a frequency of approximately 0.5 Hz, and a duration of 2 min.
- Determination of preliminary tapped volume ( $V_{St}$ ).
- Further tapping of the powder for 1 min and re-determination of the compacted volume ( $V_{St}$ ).
- Tapping for a minute and repeat until  $V_{St}$  is constant (about 1-2 ml exactly).
- Calculate the HR value following Equation 1:

$$HR = [(m_1 - m_0) \text{ in g} / V_{St} \text{ ml}] / [(m_1 - m_0) \text{ in g} / 100 \text{ ml}] = 100 \text{ ml} / V_{St} \quad (1)$$

- This procedure should be repeated 3x for each powder (each done with a new powder sample).

## 2.2 SLS powder material for HR determination

In this Round Robin study, the following materials are considered to be tested with the described method:

- Duraform DF - virgin powder
- Duraform DF – used powder
- PA 2200 – virgin powder
- irpd research powder - improved flowability

The aging status of the powders was characterised by Melt Volum Rate (MVR) measurements (see Table 2). All powder materials were provided by inspire AG, irpd (350 ml per sample) in order to be sure that every participant uses the same powder. Each sample has to be measured tree times by each contributor. All results were collected by inspire AG, irpd and evalated regarding the described statistics (chapter 1.3).

## 2.3 HR Results

The following results (Table 2, Figure 4, 5, 6 and 7) were obtained by statistical calculation (DIN 38402-45) of the single results of the 9 participants. All involved laboratories remain anonymous and are encoded from number 1 to 9. Table 2 summarizes the main statistical values for the 4 powder samples.

Table 2. Summary of the HR result obtained by Round Robin

Material	MVR g/10min	HR Test Results		
		Hampel	$S_r$	$S_R$
DF virgin	> 70	1.148	0.006	0.028
DF used	< 15	1.225	0.008	0.023
PA 2200 virgin	> 60	1.185	0.008	0.025
IrpD R-powder	----	1.116	0.004	0.048

Figure 4, 5, 6 and 7 depicts the laboratory results in comparison to each other. The calculated mean value ('Hampel estimator') is given in the Figures as well as the range of  $\pm 2 \cdot S_R$  (dotted line). The limit of  $\pm 2 \cdot S_R$  is used as a confidence interval. If the results of a participant are within this limit it can be regarded as 'good result' fully in accordance with the Round Robin Test expectations.

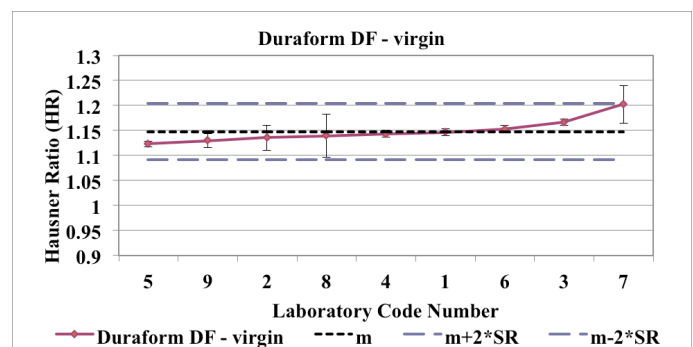


Figure 4. Summary of results of 9 labs for Duraform DF virgin

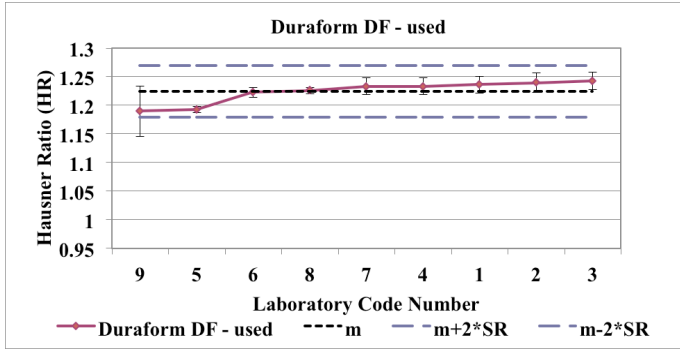


Figure 5. Summary of results of 9 labs for Duraform DF used

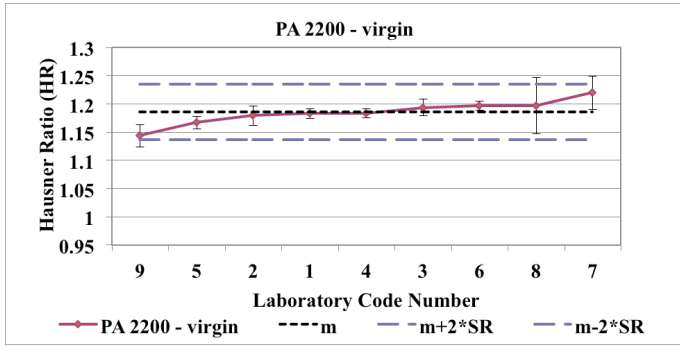


Figure 6. Summary of results of 9 labs for PA 2200 virgin

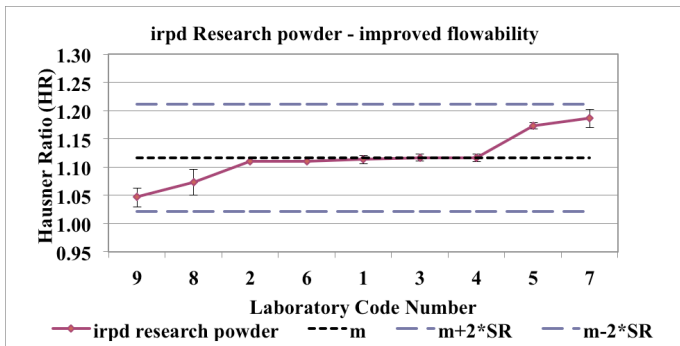


Figure 7. Summary of results of 9 labs for Irpd research powder

In Figure 8 and 9 the results of Table 2 are summarized in an optical manner for better estimation. The mean values for HR for the 4 different materials are compared to each other and supplemented with  $s_r$  (Figure 8) and  $s_R$  (Figure 9). A clear difference of HR values with significant distinguished values is obvious.

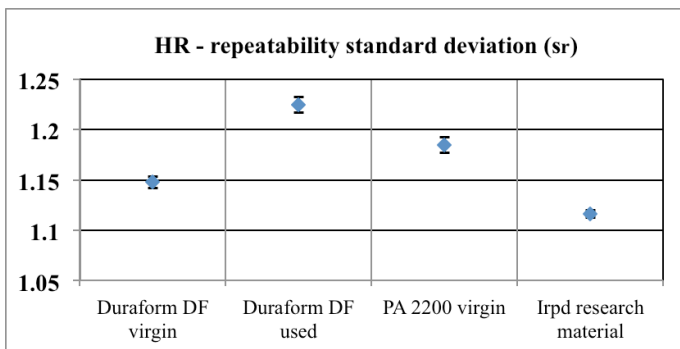


Figure 8. Materials of the round Robin in comparison with  $s_r$

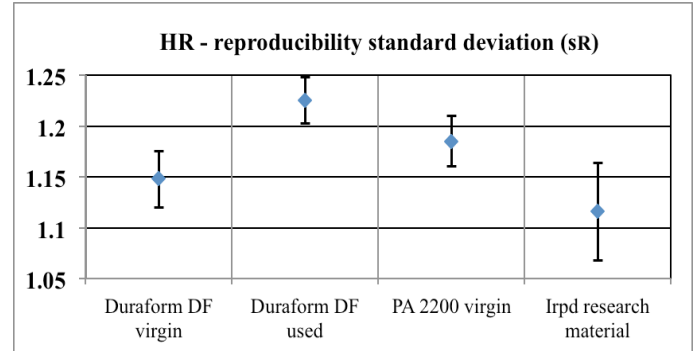


Figure 9. Materials of the round Robin in comparison with  $s_R$

## 2.4 Discussion of results

From Table 2 and the optical depiction of HR values in Figure 8 and 9 it can be derived that the 4 materials can be easily distinguished regarding flowability with this effortless method. It is surprising that simply using a plastic measuring cylinder materials widely used in SLS business can be discriminated. Not only that used (aged) Duraform DF SLS powder has a significant higher HR-value (1.225) than all others, also virgin Duraform DF (HR = 1.148) and PA 2200 (HR = 1.185) can be statistically significant differentiated.

The results for ‘Duraform DF – used’ in comparison to ‘DF- virgin’ indicate that it might be possible to access the aging status of SLS powder by HR measurements. This could be a new approach to qualify SLS powders regarding usability prior to mixing for production. Today some service bureaus use MVR measurements for this purpose, but an alternative method would be interesting, as MVR needs sophisticated equipment and has some restrictions especially for PA12 polymers.

## 3 SUMMARY

The present investigation describes a Round Robin Test comparison of 4 different SLS materials regarding powder flowability. The determination of ‘Hausner Ratio (HR)’ was performed from 9 different qualified SLS laboratories and users what means a sufficient number of data sets for significant conclusions. A robust statistical appraisal was used to achieve results for median (Hampel estimator),  $s_r$  (standard deviation of repeatability) and  $s_R$  (standard deviation of reproducibility).

With the obtained values it could be demonstrated that this simplified procedure of determining bulk and tap density is adequate to qualify and distinguish between different SLS powders. Thus, this simple method could be a promising approach to qualify PA12 SLS powders regarding their aging status and help SLS users to evaluate their powders prior to mixing. Further investigations with powders in different aged conditions are necessary and envisioned.



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